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IS 1448-30 (1970): Methods of test for petroleum and its products, Part 30: Sediment in crude and fuel oils by extraction [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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IS : 1448 [P : 30] - 1970
Adopted : *28 December 1970*

(Reaffirmed 2003)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 30]

SEDIMENT IN CRUDE AND FUEL OILS BY EXTRACTION

(First Revision)

Second Reprint DECEMBER 1983

UDC 665·5 : 543·86

Adapted from the joint publication ASTM Designation D473-65 and IP Designation 53/66.

[In this revision, the extraction solvent has been changed to toluene and the precision limits of the test results have been modified. The title has also been changed in order to align it with the corresponding joint ASTM-IP standard from where it has been adapted.]

1. SCOPE

1.1 This method prescribes the determination of the sediment in crude petroleum and fuel oils by extraction with toluene.

2. OUTLINE OF THE METHOD

2.1 The sample is taken in a refractory thimble and is extracted with hot toluene until the residue reaches constant weight. The weight of residue, calculated in percent is reported as ' sediment by extraction '.

3. APPARATUS

3.1 The extraction apparatus comprising the following parts assembled as shown in Fig. 1 and 2 is required.

3.1.1 *Extraction Flask* — A wide-mouthed conical flask of one litre capacity.

3.1.2 *Condenser* — A metal coil approximately 25 mm in diameter and 50 mm in length attached to and with the ends projecting through a lid of sufficient diameter to cover the neck of the flask as shown in Fig. 1 and 2.

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The coil shall be formed of metal or alloy tubing having an outside diameter of 8 mm and wall thickness of 1·5 mm and composed of tin or tin-coated copper or brass. The tin-coating shall have a minimum-thickness of 0·075 mm. The exposed surface of the coil for cooling purposes is about 115 sq cm.

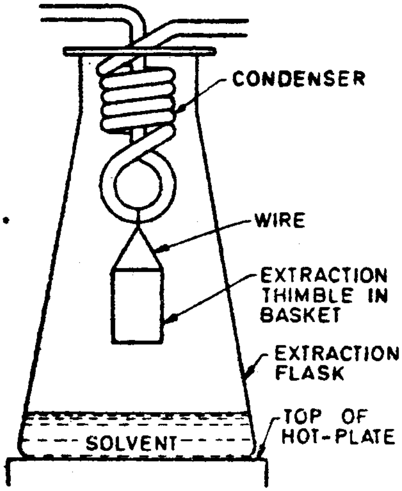

FIG. 1 EXTRACTION APPARATUS FOR DETERMINATION OF SEDIMENT

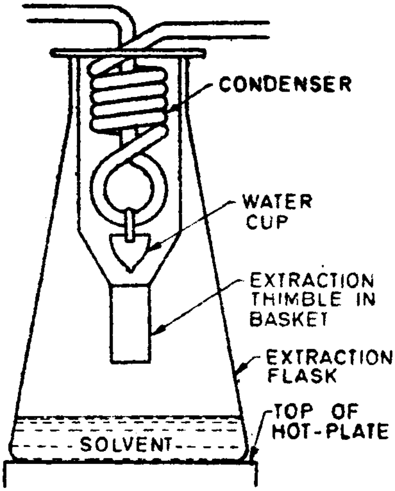

FIG. 2 EXTRACTION APPARATUS SHOWING WATER CUP IN POSITION

3.1.3 *Extraction Thimble* — A porous grade refractory thimble*, 25 mm in diameter, 70 mm in height, and of weight 15 to 17 g. The thimble shall be suspended from the condenser coil by means of a basket so that it hangs approximately 45 mm above the extracting solvent and 45 mm below the condenser coil.

3.1.3.1 For referee tests, a new extraction thimble should be used. For routine tests, thimbles may be used for a number of successive determinations on different samples, the extraction to constant weight for one determination being considered as the preliminary extraction for the succeeding determination. When the accumulation of sediment becomes excessive, the combustible portion shall be removed by heating to a dull red heat preferably in an electric furnace and the thimble shall be subjected to a preliminary extraction before being used for another determination.

3.1.4 *Water Cup* — It shall be used when testing a material having a high water content. The cup shall be made of glass, conical in shape, approximately 20 mm in diameter and 25 mm deep, and have a capacity of approximately 3 ml. It shall have a glass hook so fused on the rim at one side that when hung on the condenser, the cup hangs with its rim reasonably level.

3.1.5 *Thimble Basket* — A corrosion resistant basket made of platinum, stainless steel, nickel-chromium alloy or similar material for supporting the thimble. The thimble basket may be hung from the underside of the condenser lid as shown in Fig. 1 and 2 or from the condenser coil and shall be designed accordingly.

3.1.6 *Source of Heat* — It shall be suitable to vaporize toluene, such as a steam-bath or electric hot-plate.

4. REAGENT

4.1 Toluene — Nitration grade (*see* IS : 537-1967†).

CAUTION: Toluene is toxic. Take precautions to avoid breathing toluene vapours (*see also* IS : 4644-1968‡).

5. PROCEDURE

5.1 Before using a new thimble, rub its outer surface with fine sandpaper and remove all loosened material with a stiff brush. Give thimble a preliminary extraction with toluene allowing the solvent to drip from the thimble for at least one hour. Then dry the thimble for one hour at a temperature of 115 to 120°C, cool in a desiccator without desiccant, for one hour and weigh to the nearest 0·1 mg. Repeat the extraction until the weights of the thimble after two successive extractions do not differ by more than 0·2 mg.

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*Alundum/fused alumina thimbles listed as No. 5163RA98 by the Norton Co, Worcester 6, Mass, have proved to be satisfactory.

†Specification for toluene, pure, nitration grade (*first revision*).

‡Code of safety for benzene, toluene and xylene.

5.2 Place an estimated 10 g portion of the material in the thimble as soon as possible after the material has been thoroughly mixed. Do not attempt to adjust this estimated 10 g portion to any exact predetermined amount. Weigh to the nearest 0·01 g. Place the thimble in the extraction apparatus and extract with toluene for 30 minutes after the solvent dropping from the thimble is colourless. Adjust the rate of extraction so that the surface of the mixture of oil and toluene in the thimble does not rise higher than a point within 20 mm of the top.

5.3 When testing samples having high water contents, the water cup shall be used (*see* Fig. 2). In this procedure, any water in the material is removed as its toluene azeotrope and collected in the water cup, where it separates into two phases. The toluene layer overflows into the thimble. If the cup becomes full of water, cool the apparatus slightly and empty the cup.

5.4 After the extraction is completed, dry the thimble for one hour at 115 to 120°C. Cool in a desiccator without desiccant for one hour and weigh to the nearest 0·2 mg.

5.5 Repeat the extraction, allowing the solvent to drip from the thimble for 60 to 75 minutes; dry the thimble, cool and weigh as in **5.4**. Repeat the extraction for further one-hour period, if necessary, until the weights of the dried thimble and sediment do not differ by more than 0·2 mg after two successive extractions.

6. CALCULATION AND REPORTING

6.1 Calculate the weight of the sediment as percentage of the original sample and report it to the nearest 0·01 percent as ' sediment by extraction '.

7. PRECISION

7.1 Results of duplicate tests shall not differ by more than the following amounts:

Repeatability	*Reproducibility*
$0{\cdot}017 + 0{\cdot}255A$	$0{\cdot}033 + 0{\cdot}255A$

where

A = average result in percent weight.

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